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<b>(51) International Patent Classification <sup>6</sup> :</b> <b>C01B 33/32, B01F 3/00</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 98/15499</b> <b>(43) International Publication Date:</b> 16 April 1998 (16.04.98)
<b>(21) International Application Number:</b> PCT/US97/17495 <b>(22) International Filing Date:</b> 25 September 1997 (25.09.97)  <b>(30) Priority Data:</b> 60/028,008                      8 October 1996 (08.10.96)                      US  <b>(71) Applicant (for all designated States except US):</b> CORNING INCORPORATED [US/US]; 1 Riverfront Plaza, Corning, NY 14831 (US).  <b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only):</b> DASHER, David [US/US]; 137 Field Street #45, Corning, NY 14830 (US). MASCHMEYER, Richard, O. [US/US]; 10287 Powderhorn Drive, Corning, NY 14830 (US).  <b>(74) Agent:</b> HERZFELD, Alexander, R.; Corning Incorporated, Patent Dept., SP FR 02-12, Corning, NY 14831 (US).		<b>(81) Designated States:</b> AU, BR, CA, CN, ID, JP, KR, MX, RU, US, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> METHOD OF INHIBITING GELLING OF SILOXANE FEEDSTOCKS AND A GEL INHIBITED FEEDSTOCK  <b>(57) Abstract</b>  A method of inhibiting the gelling of a silica forming siloxane feedstock and the gelling inhibited silica feedstock which includes an end capping compound. The method of inhibiting the gelling of a siloxane feedstock during the silica glass manufacturing process includes end capping the siloxane feedstock prior to converting the siloxane feedstock into silica.		

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## METHOD OF INHIBITING GELLING OF SILOXANE FEEDSTOCKS AND A GEL INHIBITED FEEDSTOCK

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### FIELD OF THE INVENTION

The present invention relates to the formation of silica. More particularly, the present invention relates to a silicon containing feedstock for use in manufacturing silica and a method of treating feedstocks used in the formation of silica.

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Various processes are known in the art that involve the production of silica glass from vaporous reactants. Such processes require a feedstock containing silicon, a means for transporting the feedstock and an oxidant to a reaction site, and a means of decomposing the feedstock in order to convert the feedstock into silica. An apparatus used in such a process usually consists of a liquid feedstock reservoir, a means of converting said liquid feedstock into a vapor and delivering said feedstock vapor to a conversion site burner wherein the feedstock vapor is converted by flame hydrolysis into silica soot. Silica soot produced by such a burner is then collected on a deposition surface. Such collected silica may be simultaneously or subsequently heat treated to form a fused, non-porous, transparent, high purity silica glass article.

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### BACKGROUND OF THE INVENTION

Historically, silicon tetrachloride ( $\text{SiCl}_4$ ) has been used as the main silicon containing liquid feedstock that is converted into silica. The disadvantages of using  $\text{SiCl}_4$  is that the

conversion to silica results in the production of environmentally unsafe  $\text{HCl}$  as a byproduct. As an alternative to using  $\text{SiCl}_4$  as a feedstock, silica may be formed from halide-free, silicon-containing feedstocks such as siloxanes. U.S. Patent No. 5,043,002 to Dobbins et al., the disclosure of which is hereby incorporated by reference, discloses the use of such siloxanes as feedstocks. U.S. Patent No. 5,043,002 to Dobbins et al. discloses the benefits of using polyalkylsiloxanes such as polymethylsiloxanes, and in particular polymethylcyclotetrasiloxane (OMCTS) as the preferred silicon-containing feedstock, in the formation of silica.

It has been found that use of such cyclic siloxanes, and in particular octamethylcyclotetrasiloxane (OMCTS), in the manufacture of silica poses problems caused by the siloxanes' tendency to polymerize when exposed to elevated temperatures. Such cyclic siloxane feedstocks are sensitive to cracking and polymerization during vaporization and delivery to a decomposition site. When exposed to the excessive temperatures of vaporization and delivery, the cyclic structure of octamethylcyclotetrasiloxane may open up. Such opened siloxane rings will join or polymerize with other siloxane species to form higher weight siloxane species. Such polymerized higher weight siloxane species normally have boiling points exceeding the vaporizing and delivery temperatures of the silica manufacturing process and are thus deposited during vaporizing and delivery as troublesome gels. This gelling of the siloxane feedstock causes manufacturing difficulties due to the clogging and restriction of the feedstock flow during storage, vaporization, delivery and decomposition. Such gelling tends to foul heat transfer surfaces and plug the piping of delivery systems, leading to disturbance of the silica manufacturing process. Such gel formation creates great difficulty in controlling, monitoring and metering the amount and rate at which a silicon-containing feedstock is utilized in a silica manufacturing process.

### SUMMARY OF THE INVENTION

Accordingly, the present invention is directed to a silica forming feedstock which is resistant to gelling and to a method of inhibiting the gelling of siloxane feedstocks. To achieve these and other advantages and in accordance with the purpose of the invention,

as embodied and broadly described, the invention inhibits the gelling of siloxane feedstocks during the silica manufacturing process by end capping the siloxane feedstock. In another aspect, the invention includes a silica forming feedstock of polyalkylsiloxane and an end capping compound which inhibits the polymerization of the polyalkylsiloxane.

It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory and are intended to provide further explanation of the invention as claimed.

The accompanying figures are included to provide a further understanding of the invention and are incorporated in and constitute a part of this specification, illustrate an embodiment of the invention and, together with the description, serve to explain the principles of the invention.

#### BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a Fourier Transform Infrared (FTIR) Spectra Analyses of an embodiment and components of the invention.

FIG. 2 is a Fourier Transform Infrared (FTIR) Spectra Analyses of an embodiment and components of the invention.

FIG. 3 is a Fourier Transform Infrared (FTIR) Spectra Analyses of an embodiment and components of the invention.

FIG. 4 is a Gel Permeation Chromatography (GPC) Analyses of an embodiment and components of the invention.

#### DESCRIPTION OF THE INVENTION

The invention is directed to a method of inhibiting the gelling of siloxane feedstocks, used in manufacturing and forming silica, by end capping the siloxane

feedstock. By end capping the siloxane feedstock, the polymerization of the siloxane feedstock is restrained. By restraining the polymerization of the siloxane feedstock, the formation of gels during the silica producing process is inhibited.

The invention discloses a polyalkylsiloxane silica forming feedstock which includes an end capping compound which inhibits polymerization of the feedstock and formation of gels during the silica manufacturing process.

Reference will now be made in detail to the present preferred embodiment of the invention.

In using the phrase end capping, the inventors mean that the normally polymerizing ends of the siloxane compound are preferentially reacted with a compound which prevents these normally polymerizing ends from polymerizing with other siloxanes and forming higher molecular weight siloxane polymers. In accordance with the invention, the kinetics governing the preferential reaction of the end capping compounds with the normally polymerizing ends are quicker than the kinetics of the siloxane polymerization reaction.

Octamethylcyclotetrasiloxane is a preferred siloxane containing halide free feedstock. Octamethylcyclotetrasiloxane is decomposed to form silica which is utilized in the fabrication of optical fibers, planar waveguides, lightwave optical circuits, bulk fused silica, high purity fused silica members and other silica glass based products. Although the use of an octamethylcyclotetrasiloxane feedstock is preferred, it has disadvantages due to the octamethylcyclotetrasiloxane ring structure's tendency to open up during vaporization from a liquid and delivery of the vaporized feedstock to the decomposition site where it is converted to silica ( $\text{SiO}_2$ ). Octamethylcyclotetrasiloxane molecules may open up and subsequently polymerize with other octamethylcyclotetrasiloxane molecules under the temperature and other environmental conditions, including operating pressures, that are present in the silica manufacturing process, particularly the steps of vaporization of the feedstock, delivery of the vaporized feedstock to the decomposition site, and decomposition of the feedstock into silica. Such polymerization converts the octamethylcyclotetrasiloxane molecules into higher molecular weight siloxane polymers. These higher molecular weight siloxane polymers

are not very volatile and tend to form gels which plug the vaporization, delivery, and decomposition systems of the silica manufacturing process.

The inventors have discovered that such polymerizing and gelling of the octamethylcyclotetrasiloxane feedstock can be inhibited and prevented by introducing an end capping chemical compound into the liquid feedstock before the feedstock is vaporized. By introducing an end capping chemical compound into the liquid feedstock, octamethylcyclotetrasiloxane molecules which open up are end capped and prevented from polymerizing with other present siloxanes. The end capping compound will react with the normally polymerizing octamethylcyclotetrasiloxane opened ring ends to form an octamethylcyclotetrasiloxane end capped complex which will not polymerize with other siloxanes present in the silica manufacturing process.

The presently preferred end capping compound of the invention is triethyl borate. Triethyl borate introduced into the feedstock is able to effectively end cap the octamethylcyclotetrasiloxane thus preventing the polymerizing and gelling of this siloxane feedstock. Triethyl borate is preferred in that it can serve as a boron source compound for boron oxide which is utilized as a dopant in many silica compositions and products such as optical waveguides, planar waveguides, and lightwave optical circuits. The beneficial end capping properties of triethyl borate can be attributed to boron which is electron deficient which makes the triethyl borate molecule very reactive toward nucleophilic reagents, which contain atoms such as oxygen which have lone pairs of electrons.

In addition, triethyl borate's boiling point and vapor pressure are compatible with those of octamethylcyclotetrasiloxane. Triethyl borate has a boiling point of 117-118°C. Having such a boiling point over 100°C is beneficial since it allows the triethyl borate to be present both as a vapor and a liquid at above 100°C temperatures and pressures typically utilized in the silica manufacturing process. Normally a temperature of 120°C or higher is needed to vaporize an octamethylcyclotetrasiloxane liquid feedstock. Octamethylcyclotetrasiloxane ring opening, which without end blocking leads to gel formation, typically begins to occur in the 150°C to 200°C temperature range for the commercially available high purity grade octamethylcyclotetrasiloxane used in the formation of high purity silica.

Triethyl borate's compatibility with octamethylcyclotetrasiloxane in terms of solubility and boiling points allows this end capping compound to be present and effective in the liquid and vapor states experienced by octamethylcyclotetrasiloxane molecules during vaporization and delivery.

5 It is preferred to introduce the liquid end capping compound into the siloxane feedstock when the siloxane feedstock is in the liquid state. It is preferred to introduce and incorporate liquid triethyl borate into the liquid octamethylcyclotetrasiloxane before the feedstock is vaporized. Due to the compatibility between triethyl borate's and octamethylcyclotetrasiloxane's boiling points, the end capping compound is present and  
10 end capping may occur throughout the silica manufacturing steps where octamethylcyclotetrasiloxane is susceptible to polymerization and gelling.

When using triethyl borate as an end capping compound, the triethyl borate combines with the opened ring octamethylcyclotetrasiloxane molecule to form a triethyl borate-octamethylcyclotetrasiloxane complex which is vaporizable along with the  
15 unopen octamethylcyclotetrasiloxane molecules. Such end capped triethyl borate-octamethylcyclotetrasiloxane complexes are resistant to polymerizing and gelling. After vaporization, these end capped triethyl borate-octamethylcyclotetrasiloxane complexes travel through the vaporized feedstock delivery system and are then converted to silicon dioxide and boron oxide, without troublesome gelling.

20 End capping the siloxane feedstock with triethyl borate is preferred in the manufacturing of planar waveguides, particularly when the siloxane containing feedstock is converted to silica by flame hydrolysis. In the manufacturing of planar waveguides, core and clad layers are formed which guide the light. The core and clad layers are formed from silica glass compositions that differ from one another in order to provide an  
25 appropriate difference in refractive index. The inventors utilized the inventive method of end capping a siloxane feedstock in the manufacturing of such a clad glass layer.

A clad glass liquid feedstock containing approximately 4.30 wt% trimethyl phosphate, 12.80 wt% triethyl borate and 82.70 wt% octamethylcyclotetrasiloxane was mixed together and pumped to a vaporizer. This triethyl borate end capped siloxane  
30 feedstock was vaporized, and the vaporized feedstock was delivered to a burner where it was converted into silica.



Gel Permeation Chromatography (GPC) and Fourier Transform Infrared (FTIR) analysis were performed on this borate end capped siloxane containing feedstock, which showed that polymerization of the feedstock did not occur during vaporization. In these analyses, one liter liquid samples were evaporated down to approximately .5 ml to 1 ml of concentrated liquid. During the evaporation of the clad glass liquid feedstock a precipitate formed. The concentrated liquid clad glass feedstock was separated from the precipitate by dissolving it in toluene. The toluene was then evaporated off. The precipitate was determined to be almost entirely water soluble, indicating that it did not contain high molecular weight siloxanes. FIG. 1 shows the FTIR spectra of individual components octamethylcyclotetrasiloxane 20, triethyl borate 21, trimethyl phosphate 22, and a vaporized 4.30 wt% trimethyl phosphate, 12.80 wt% triethyl borate, and 82.70 wt% octamethylcyclotetrasiloxane clad glass feedstock mixture 23 in descending order. The spectra of the individual components of triethyl borate, trimethyl phosphate, and octamethylcyclotetrasiloxane exhibit characteristic group frequencies consistent with their respective structures. The characteristic group frequencies of all three components can be seen in the vaporized concentrated liquid clad glass feedstock mixture as shown by spectra 23 of FIG. 1.

FIG. 2 compares the FTIR spectra of octamethylcyclotetrasiloxane 24, the vaporized concentrated liquid clad glass feedstock mixture GPC Sample 25, vaporized octamethylcyclotetrasiloxane concentrate GPC Sample 26, and polymerized octamethylcyclotetrasiloxane 27 in descending order. Evidence of the siloxane in the vaporized concentrated liquid clad glass feedstock mixture are the bands in the FTIR spectrum at  $810\text{ cm}^{-1}$  (Si-CH<sub>3</sub>),  $1070\text{ cm}^{-1}$  (Si-O-Si),  $1254\text{ cm}^{-1}$  (Si-CH<sub>3</sub> sym. def.) and 2850-2980 cm (CH stretch). The bands between 1320-1640  $\text{cm}^{-1}$  are due to triethyl borate and trimethyl phosphate with additional borate bands between 740-780  $\text{cm}^{-1}$ .

FIG. 3 compares the FTIR spectra of the clad glass feedstock mixture 28, the vaporized concentrated liquid clad glass mixture GPC Sample 29, the clad glass feedstock mixture solid precipitate 30, and the clad glass feedstock mixture solid precipitate after exposure to air 31, in descending order. Evidence of a siloxane borate complex can be seen in the clad glass feedstock mixture solid precipitate FTIR spectra 30. The bands at  $810\text{ cm}^{-1}$ ,  $1076\text{ cm}^{-1}$ , and  $1254\text{ cm}^{-1}$  indicate the presence of a

siloxane. The broad band at  $1450\text{ cm}^{-1}$  indicates a borate ester or borate. The FTIR spectra of the clad glass feedstock mixture solid precipitate after exposure to air 31, exhibits characteristic group frequencies consistent with a borate ester with no siloxane bands, indicating that the precipitate is largely a borate compound.

5        FIG. 4 shows the Gel Permeation Chromatography (GPC) traces of an octamethylcyclotetrasiloxane concentrate 32, a vaporized octamethylcyclotetrasiloxane concentrate 33, and the vaporized concentrated liquid clad glass mixture 34. Higher weight siloxanes appear on the left hand side of FIG. 4. Trace 32 shows the GPC trace obtained for the high molecular weight siloxanes of a typical  
10        octamethylcyclotetrasiloxane feedstock. Trace 33 shows the GPC trace of the high molecular weight siloxanes obtained from vaporized octamethylcyclotetrasiloxane. Trace 34 shows the GPC trace of the vaporized concentrated liquid clad glass mixture which is flat in the high molecular weight siloxane region on the left hand side of FIG. 4 but contains the low molecular weight cyclics on the right hand side as represented by  
15        the small double peaks. From these GPC traces it was determined that the typical unvaporized octamethylcyclotetra-siloxane had a high molecular weight siloxane content of approximately 100 ppm, the vaporized octamethylcyclotetrasiloxane had a high molecular weight siloxane content of approximately 63.5 ppm, and the high molecular weight siloxane content of the vaporized liquid clad glass mixture was very low and  
20        unmeasurable. These FTIR spectra and GPC traces show the ability of borate to end cap the opened ring siloxane and inhibit the polymerization of the siloxane with other siloxanes. This end capping reaction can be attributed to boron's electron deficiency, which makes the end capping molecule very reactive toward nucleophilic reagents having atoms with lone pairs of electrons, such as oxygen.

25        Although triethyl borate is the preferred end capping compound for octamethylcyclotetrasiloxane in silica forming processes used in the manufacturing of boron containing waveguides, other end capping compounds can be used to end cap siloxane feedstocks to inhibit gelling.

30        Trisopropyl borate, which has a boiling point in the range of  $134\text{--}141^\circ\text{C}$ , and tributyl borate, which has a boiling point of  $233^\circ\text{C}$ , can be used as end capping compounds for siloxane feedstocks such as octamethylcyclotetrasiloxane. Also other

boron containing compounds such as tri-tert-butyl borate, trimethyl boroxine, dichloro phenylborane, dihydroxy phenylborane, and alkyl boron chloride can be used as end capping compounds.

Also alkoxides containing other Periodic Table Group IIIA elements can be used as end capping compounds.

Particular end capping compounds should be chosen having vapor pressures or boiling points compatible with the siloxane feedstock whose polymerization and gelling is being inhibited under the conditions of the silica manufacturing process.

The end capping compound should be sufficiently soluble in the siloxane feedstock. In particular, it is beneficial to have the end capping compound present with the siloxane feedstock when the siloxane is most likely to polymerize, such as at high temperatures. In addition, end capping compounds should be chosen in view of the elements that are required to be present or absent in the formed silica. Further, the composition of the silica forming feedstock in terms of the percentage of end capping compound should be chosen in view of the percentage of elements that are required to be present or absent in the formed silica and the degree of gell formation that is to be inhibited. For example, a silica forming feedstock comprised of siloxane and 0.5 wt% to 12 wt% triethyl borate is preferred to result in a silica glass with minimal boron content while inhibiting gell formation.

It will be apparent to those skilled in the art that various modifications and variations can be made in the method and composition of the present invention without departing from the spirit or scope of the invention. Thus, it is intended that the present invention cover the modifications and variations of this invention provided they come within the scope of the appended claims and their equivalents.

## WE CLAIM:

1. A method of inhibiting the gelling of a siloxane feedstock during the manufacturing of silica, said method comprised of the steps of:

- 5 (a) obtaining a siloxane feedstock;  
(b) end capping said siloxane feedstock; and  
(c) converting said end capped siloxane feedstock to silica.

10 2. A method according to claim 1 wherein the step of end capping said siloxane feedstock includes the step of mixing an end capping compound with said siloxane feedstock wherein said end capping compound inhibits the gelling of said siloxane feedstock.

15 3. A method according to claim 1 wherein said siloxane feedstock is comprised of polyalkylsiloxane.

4. A method according to claim 1 wherein said siloxane feedstock is comprised of octamethylcyclotetrasiloxane.

20 5. A method according to claim 1 wherein the step of end capping said siloxane feedstock includes the step of introducing into said siloxane feedstock an effective amount of an end capping compound in order to inhibit polymerization of the siloxane feedstock.

25 6. A method according to claim 5 wherein the step of introducing into said siloxane feedstock an effective amount of an end capping compound includes the step of adding from about 1 wt% to 30 wt% of the end capping compound to said siloxane feedstock.

30 7. A method according to claim 5 wherein said end capping compound is comprised of an electron deficient atom.

8. A method according to claim 5 wherein said end capping compound is comprised of a Periodic Table Group IIIA atom.

5 9. A method according to claim 5 wherein said end capping compound is comprised of a boron atom.

10. A method according to claim 5 wherein said end capping compound is comprised of a borate.

10 11. A method according to claim 5 wherein said end capping compound is comprised of triethyl borate.

12. A silica forming feedstock comprised of a polyalkylsiloxane and an end capping compound which inhibits the polymerization of said polyalkylsiloxane.

15 13. A silica forming feedstock according to claim 12 wherein said polyalkylsiloxane is comprised of octamethylcyclotetrasiloxane.

20 14. A silica forming feedstock according to claim 12 wherein said end capping compound is comprised of an atom with a vacant orbital.

15. A silica forming feedstock according to claim 12 wherein said end capping compound is comprised of triethyl borate.

25 16. A silica forming feedstock according to claim 12 wherein said end capping compound has a boiling point greater than about 80°C.

30 17. A silica forming feedstock according to claim 12 wherein said end capping compound has a boiling point compatible with the polyalkylsiloxane's boiling point.

18. A silica forming feedstock according to claim 12 wherein said end capping compound has a boiling point greater than about 100°C.

5 19. A silica forming feedstock comprised of octamethylcyclo-tetrasiloxane and triethyl borate.

20. A silica forming feedstock according to claim 19 comprised of about 1 wt% to 30 wt% triethyl borate.

10 21. A silica forming feedstock according to claim 19 comprised of about 7 wt% to 18 wt% triethyl borate.

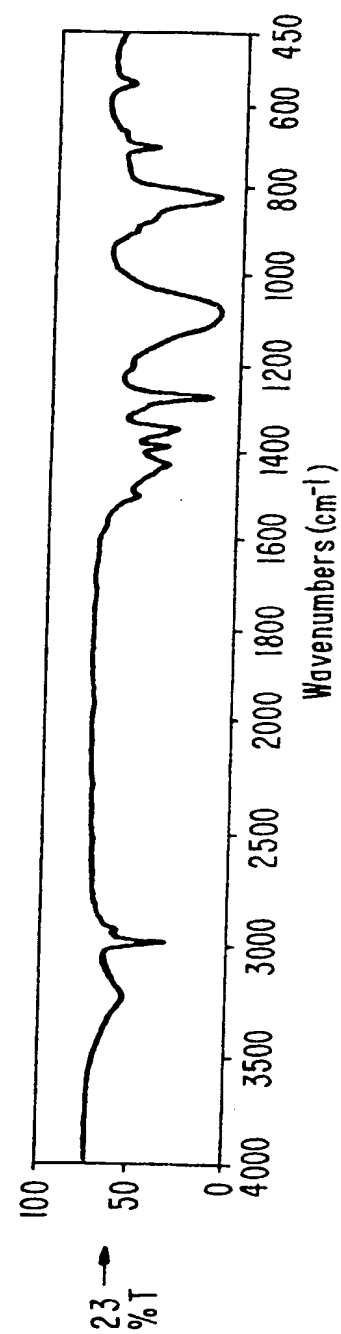
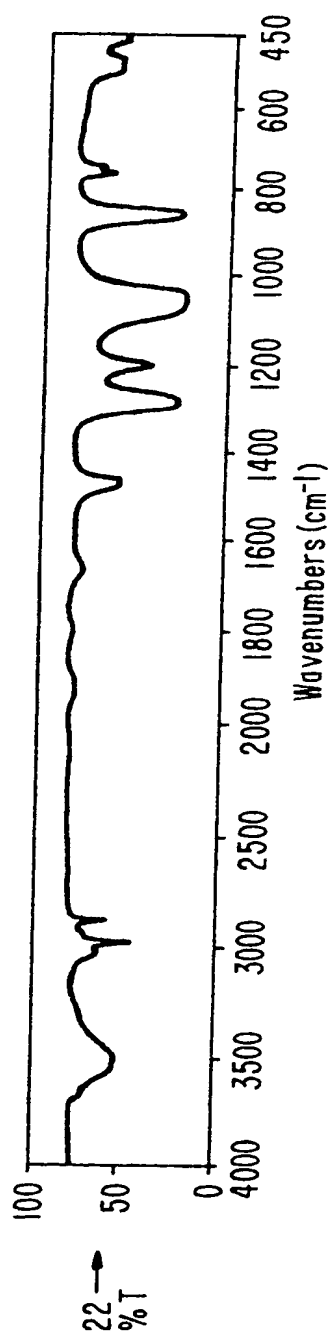
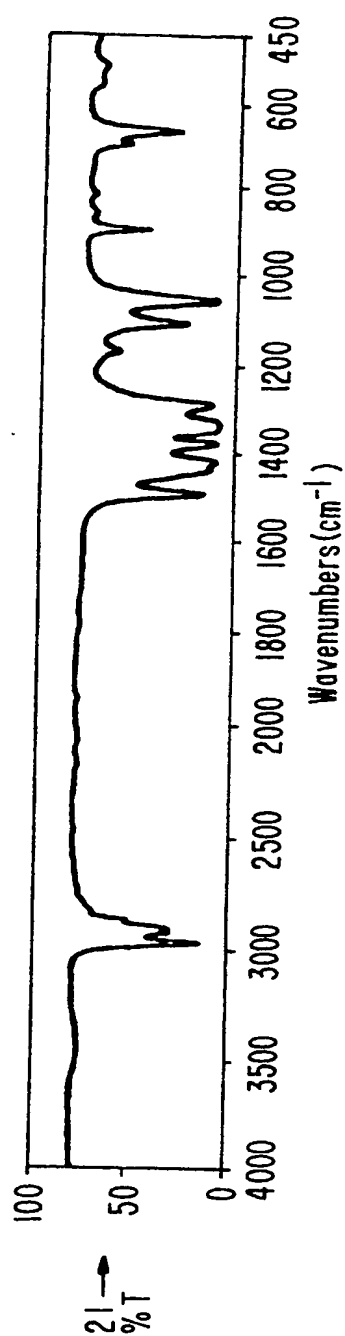
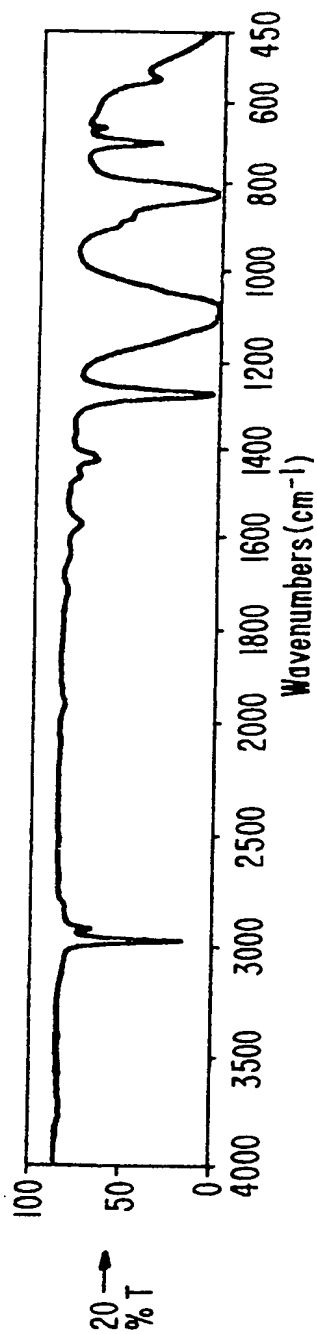
22. A silica forming feedstock according to claim 19 comprised of about 11 wt% to 14 wt% triethyl borate.

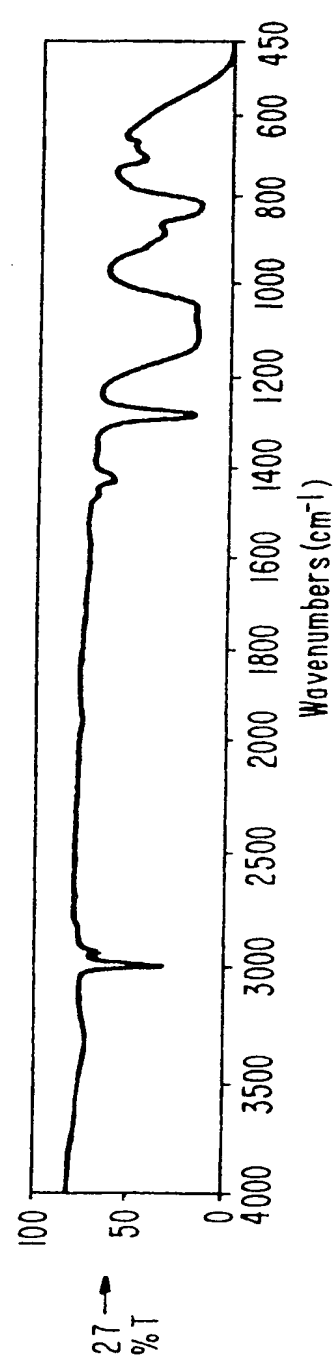
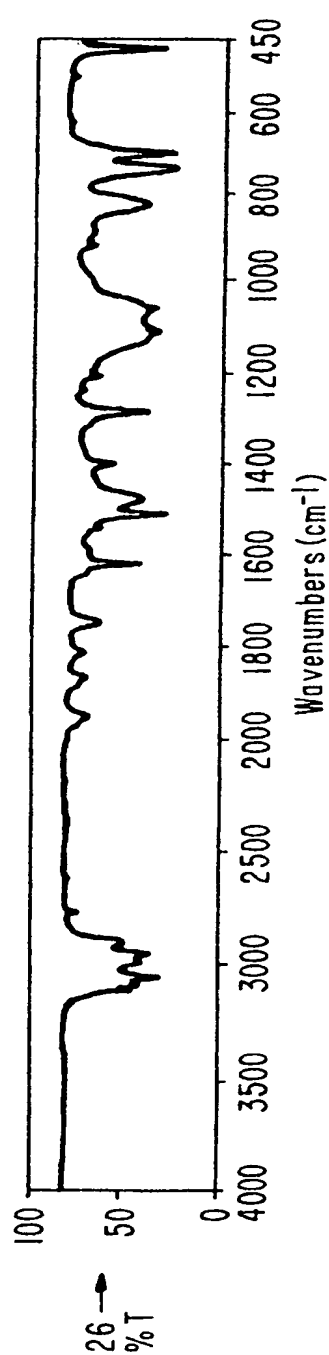
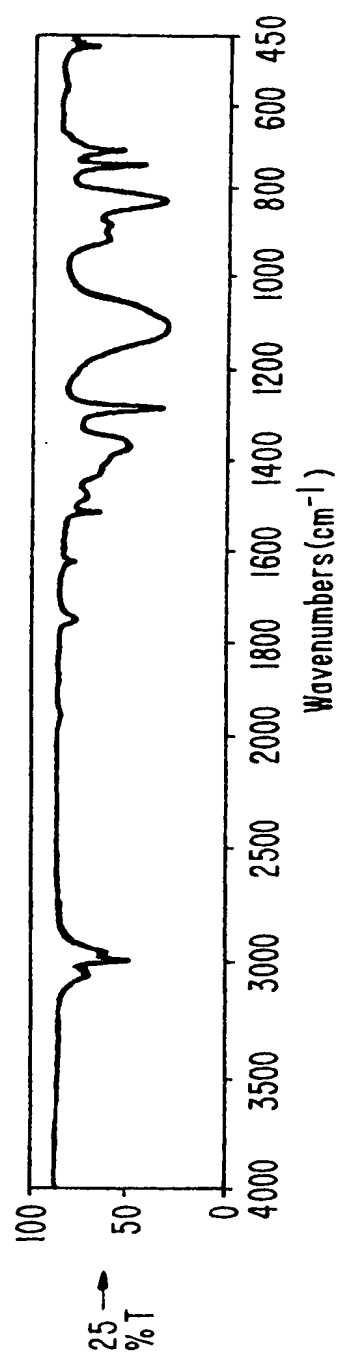
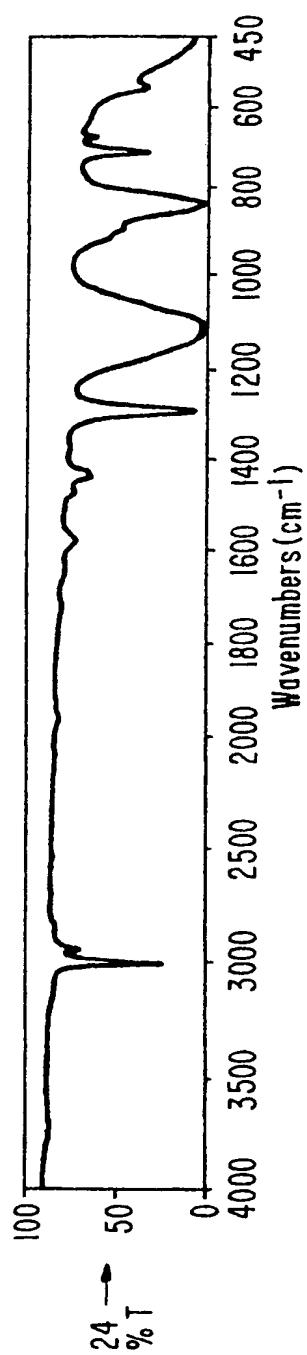
15 23. A silica forming feedstock according to claim 19 further comprised of trimethyl phosphate.

20 24. A silica forming feedstock according to claim 19 comprised of about 0.5 wt% to 12 wt% triethyl borate.

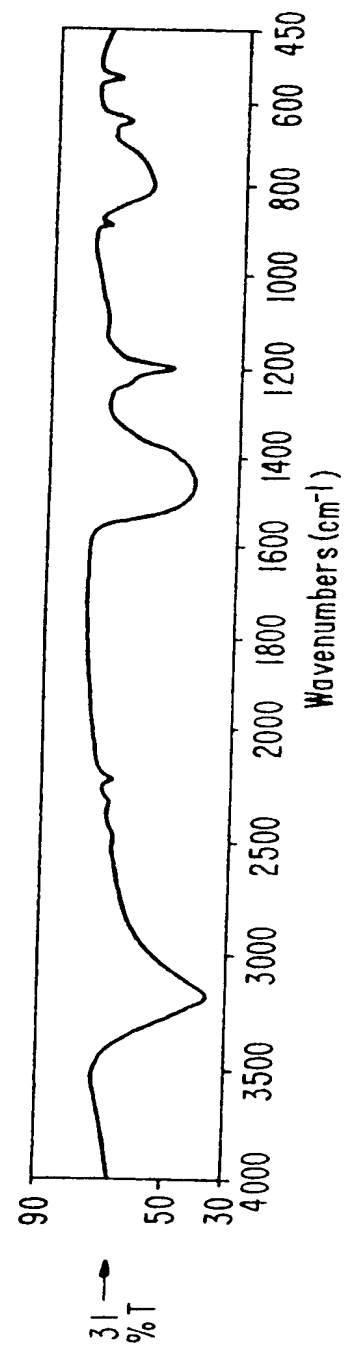
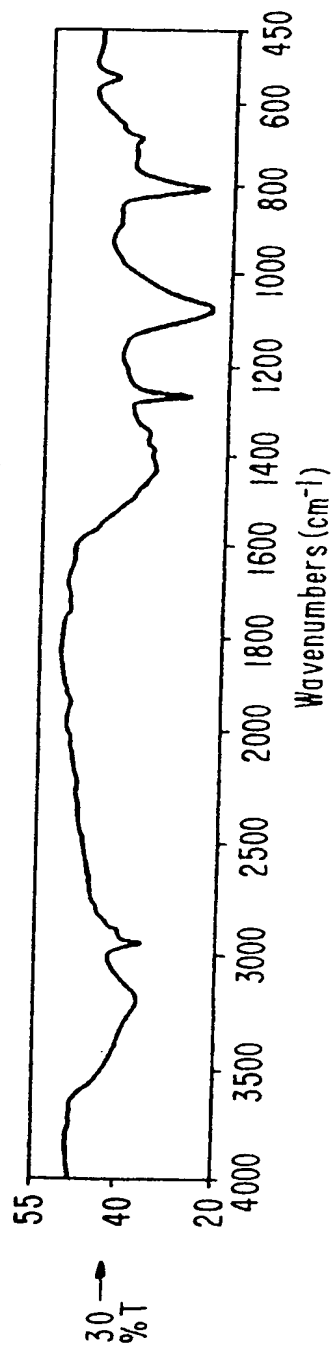
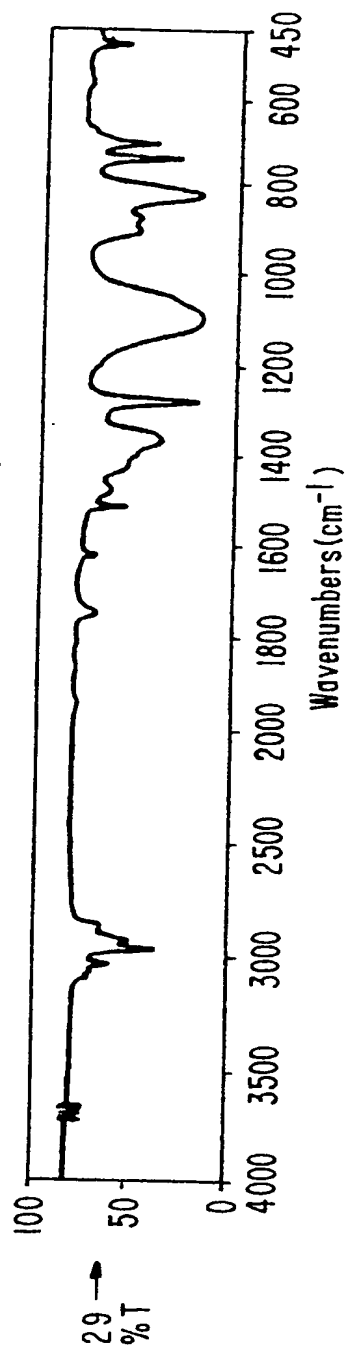
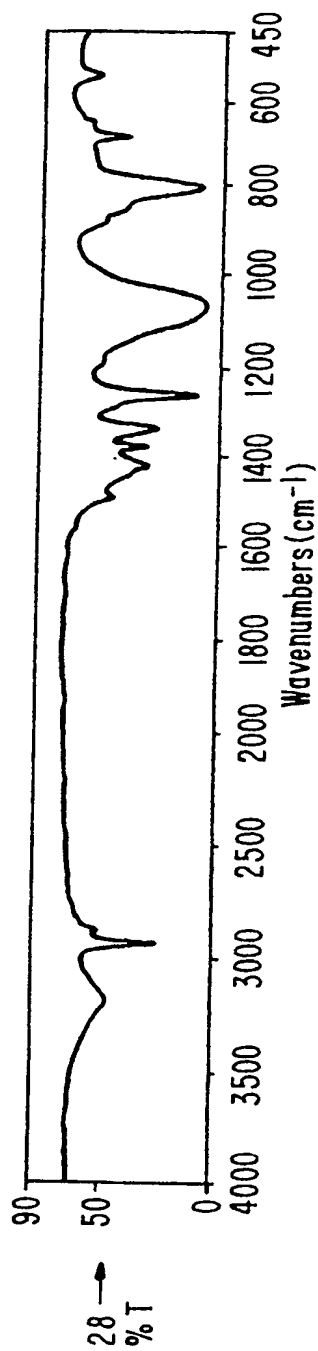
25 25. A silica forming feedstock consisting essentially of octamethylcyclotetrasiloxane and triethyl borate.

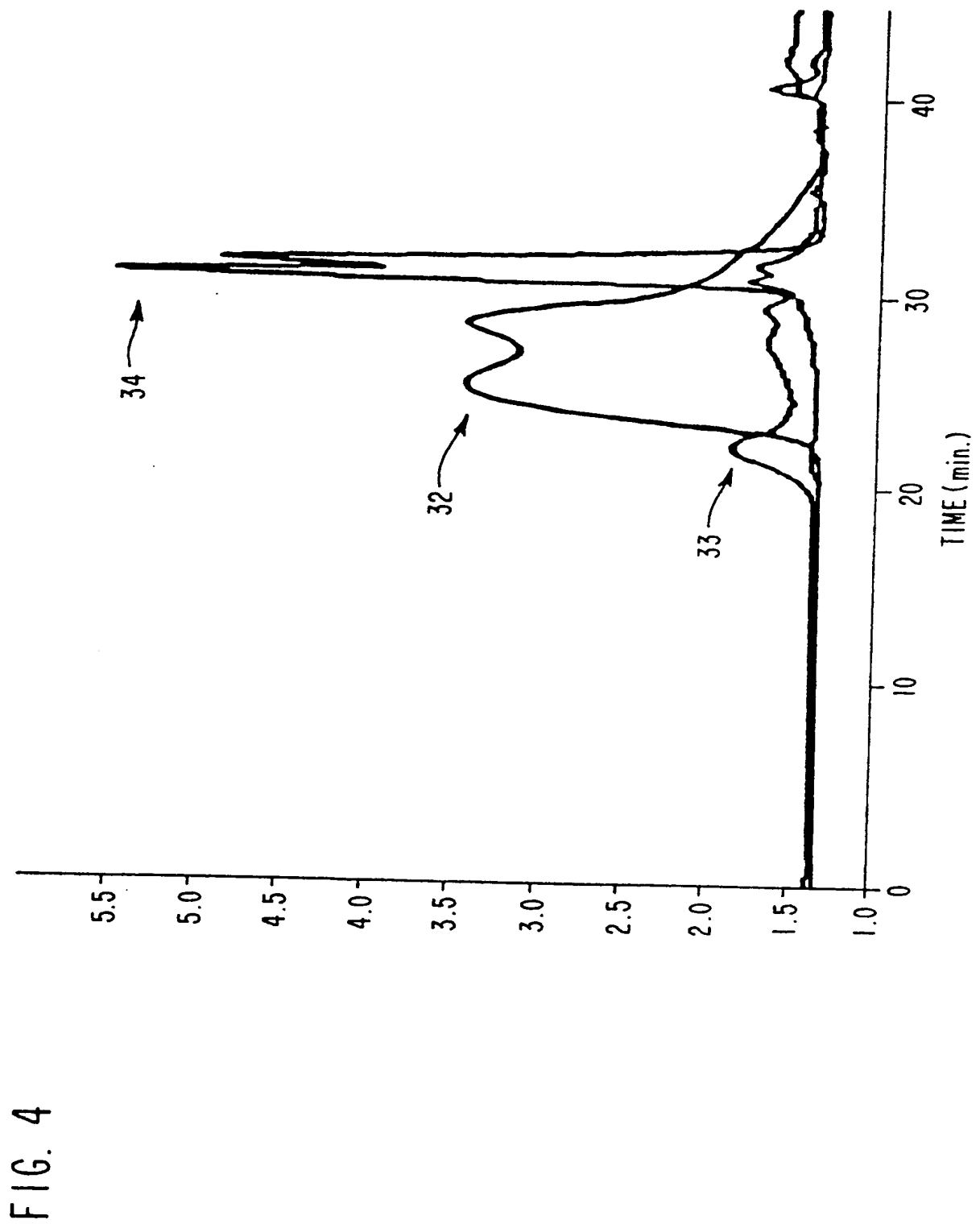
26. A method of making silica glass, said method comprised of the steps of:  
(a) obtaining a siloxane feedstock;  
(b) end capping said siloxane feedstock;  
(c) delivering said end capped siloxane feedstock to a conversion site; and  
(d) converting said end capped siloxane feedstock into silica glass.











# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US97/17495

<b>A. CLASSIFICATION OF SUBJECT MATTER</b> IPC(6) : C01B 33/32; B01F 3/00 US CL : 423/335, 337; 252/302, 318, 319 According to International Patent Classification (IPC) or to both national classification and IPC		
<b>B. FIELDS SEARCHED</b> Minimum documentation searched (classification system followed by classification symbols) U.S. : 423/335, 337; 252/302, 318, 319 Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)		
<b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
X	US 5,231,058 A (MAEDA ET AL) 27 July 1993 (27-07-93), columns 2-4	1-26
A	US 3,213,048 A (BOOT) 19 October 1965 (19-10-65), column 1	1-26
A	US 5,098,747 A (KALCHAUER ET AL) 24 March 1992 (24-03-92), column 2	1-26
<input type="checkbox"/> Further documents are listed in the continuation of Box C. <input type="checkbox"/> See patent family annex.		
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